

CLAIMS

1. A method for purifying (meth)acrylic acid obtained by catalytic or redox oxidation, of a gas substrate consisting of propane and/or propylene and/or acrolein in the case of the manufacture of acrylic acid, and of isobutane and/or isobutene and/or tert-butyl alcohol and/or methacrolein in the case of the manufacture of methacrylic acid, said gas mixture (1) mainly consisting of:
- propane and/or propylene or isobutane and/or isobutene if previously contained by the substrate;
  - final oxidation products;
  - the desired (meth)acrylic acid;
  - (meth)acrolein;
  - tertbutyl alcohol in the case of the manufacture of methacrylic acid;
  - water vapor;
  - acetic acid with, in the case of the manufacture of methacrylic acid, acrylic acid as a byproduct; and
  - heavy products of side reactions,
- according to which the reaction gas mixture (1) is sent to the bottom of an absorption column (C1) which is supplied at the top and in countercurrent with at least one heavy hydrophobic absorption solvent, to obtain:
- at the top of the column (C1) a gas stream (7) consisting of:
    - propane and/or propylene or isobutane and/or isobutene, according to whether acrylic acid or methacrylic acid is manufactured, and the products of the final oxidation of the mixture (1);
    - major quantities of water and acetic acid in the case of the manufacture of acrylic acid, or of water, acetic acid and acrylic acid in the case of the manufacture of methacrylic acid; and
    - (meth)acrolein;
  - at the bottom of said column (C1), a stream (4) consisting of:

- (meth)acrylic acid;
  - the heavy absorption solvent or solvents;
  - the heavy products of side reactions; and
  - minor quantities of acetic acid and water, in
- 5 the case of the manufacture of acrylic acid and acetic acid, acrylic acid and water in the case of the manufacture of methacrylic acid, the stream (4) issuing from the column (C1) is then sent to a separation column (C2) in which a separation
- 10 is carried out to obtain:
- at the top, a stream (5) consisting of light impurities which are sent to the bottom part of the absorption column (C1); and
  - at the bottom, a stream (6) consisting of:
- 15 - (meth)acrylic acid in solution in the absorption solvent or solvents;
- a small proportion of acetic acid in the case of the manufacture of acrylic acid and acetic acid and of acrylic acid in the case of the manufacture of
- 20 methacrylic acid;
- the heavy products of side reactions; and
  - the polymerization inhibitor or inhibitors,
- characterized in that the column (C1) is operated with a heavy solvent flow rate that is 3 to 5.6 times the
- 25 flow rate of (meth)acrylic acid in the feed gas mixture, and in that, as a separation column (C2), a rectification column is used, which is operated with a flow feed and without reflux.
2. The method as claimed in claim 1, characterized
- 30 in that the column (C2) is operated under conditions such that its distillate rate relative to the flow rate of (meth)acrylic acid introduced into the absorption column (C1) is between 0.5/1 and 4/1.
3. The method as claimed in claim 2, characterized
- 35 in that the column (C2) is operated under conditions such that its distillate rate relative to the flow rate of (meth)acrylic acid introduced into the absorption column (C1) is between 2/1 and 3/1.

4. The method as claimed in one of claims 1 to 3, characterized in that the column (C1) is operated with a solvent flow rate that is 3 to 4 times the flow rate of (meth)acrylic acid in the feed gas mixture.
- 5 5. The method as claimed in one of claims 1 to 4, characterized in that an absorption column (C1) is used comprising:
- in its lower part, at least one cooling section (S1) equipped with a system for recirculating, via an external heat exchanger (E1), part (3) of the stream (4) collected in the lower part of said section or sections (S1) to send it to the flow of said sections; and
  - in its upper part, a section (S2) for the absorption and rectification of the gas mixture (1).
- 15 6. The method as claimed in claim 5, characterized in that a section (S2) is used, in which the number of theoretical plates is 25 to 50, and preferably 30 to 45.
- 20 7. The method as claimed in one of claims 1 to 6, characterized in that the absorption is carried out in the column (C1) at atmospheric pressure or under a pressure close to atmospheric pressure, and at a solvent introduction temperature of 20 to 80°C, preferably 30 to 60°C.
- 25 8. The method as claimed in one of claims 1 to 7, characterized in that the column (C1) is operated at a bottom temperature of 50 to 120°C, particularly of 70 to 100°C.
- 30 9. The method as claimed in one of claims 1 to 8, characterized in that the column (C1) is operated at a overhead gas temperature of 40 to 70°C, particularly of 50 to 60°C.
10. The method as claimed in one of claims 1 to 9, characterized in that the reaction gases are introduced at a temperature of 100°C to 200°C, particularly 130°C to 180°C.
- 35 11. The method as claimed in one of claims 1 to 10, characterized in that one or more heavy hydrophobic

absorption solvents are used, having a boiling point above 200°C at atmospheric pressure.

12. The method as claimed in claim 11, characterized in that ditolyether is used as a heavy  
5 hydrophobic solvent.

13. The method as claimed in one of claims 1 to 12, characterized in that the absorption column (C1) is fed with one or more pure solvents and/or with one or more solvents issuing from the recycling of one or more  
10 streams obtained from the subsequent purification steps.

14. The method as claimed in one of claims 1 to 13, characterized in that the absorption is carried out in the column (C1) in the presence of at least one  
15 polymerization inhibitor, selected in particular from phenolic derivatives such as hydroquinone and its derivatives such as methyl ether of hydroquinone, phenothiazine and its derivatives, such as methylene blue, quinones, such as benzoquinone, metal  
20 thiocarbamates, such as copper dibutyldithiocarbamate, compounds with nitroso groups, such as N-nitroso-phenylhydroxylamine, amines such as derivatives of paraphenylenediamine, or N-oxyl compounds, such as 4-hydroxy-2,2,6,6-tetramethylpiperidine-N-oxyl.

25 15. The method as claimed in one of claims 1 to 14, characterized in that the gas stream (7) issuing from the top of the column (C1) is removed, partly to the reaction section, and partly to an incineration or purge step.

30 16. The method as claimed in one of claims 1 to 14, characterized in that the gas stream (7) issuing from the top of the column (C1) is sent to the bottom of a condensation section (S3) where this gas mixture is placed in intimate contact with a descending liquid  
35 stream (7') supplied at the flow of said section (S3) and consisting of the recycling of part of the bottom stream of said section (S3) previously cooled by an external heat exchanger (E4).